

A DETERGENT TEST FOR THE MILK FAT CONTENT
OF DAIRY PRODUCTS. I. MILK,
CREAM, AND ICE CREAM

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A detergent procedure for determining butterfat in milk and cream was published in 1952 (4) and identified as the BDI detergent test. Since this paper also presented a résumé of the development of detergent testing, no further literature review will be given here. The present paper describes a modified form of the test which will hereafter be referred to as the DPS (Dairy Products Section) detergent test.

In studying the accuracy of the original test as applied to milk, cream, and ice cream, it was found that minor defects occasionally marred the appearance of the fat column and that when the regular test for milk was applied to one particular brand of chocolate ice cream, it failed to yield complete separation of the fat. These findings indicated a need for additional improvement. The physico-chemical factors involved in tests with detergents have therefore been investigated further. As a result, several minor changes have been made in the test, including addition of sodium bicarbonate to the detergent-tetraphosphate reagent and slight modification of the heating and shaking procedure. These changes have helped to improve the methodology of the test and to correct occasional defects, thus improving the accuracy and appearance of the detergent test and increasing its usefulness.

This paper presents the modified method and compares the test with the Babcock and Roese-Gottlieb tests on samples of milk, cream, and ice cream.

EXPERIMENTAL

Most of the milks tested were obtained from the herd of the U. S. Department of Agriculture at Beltsville, Md. The samples included mixed

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herd milk and also milks from individual cows. Samples of homogenized milk and of ice cream were obtained from retail markets. The Babcock tests on milk and cream were conducted as described in *Official Methods of Analysis* of the A.O.A.C., 8th Ed. (1), p. 248-249, and method 2, p. 262. The Roese-Gottlieb tests were conducted with the Mojonnier equipment, as directed by the manufacturer (2). The detergent tests were conducted by the modified detergent method described below.

For the purpose of definition, the phrase "bottom to bottom" means that the measurement of the fat column was made from the bottom of the lower meniscus to the bottom of the upper meniscus; "bottom to top," from the bottom of the lower meniscus to the highest point of the upper meniscus; and "bottom to reader," from the bottom of the lower meniscus to the line formed by an added meniscus remover (reader) such as glymol.

DPS DETERGENT METHOD FOR MILK

REAGENTS

(a) *Detergent reagent*.—Dissolve 7.0 g Na tetraphosphate, 2.0 g NaHCO_3 , and 3.0 g of either Triton X-100¹ or Tergitol Dispersant NPX¹ by stirring, and dilute to 100 ml with distilled water. (Turbidity caused by heat of solution disappears on cooling to room temperature.) If the solution has been stored for more than two months at room temperature or one year at 10°C., prepare fresh solution.

(b) *Methyl alcohol*.—A.C.S., 50% by volume.

PROCEDURE

The test bottle and milk pipet are the same as specified for the official Babcock test, *Official Methods of Analysis*, 8th Ed., pp. 248-249 (1). Likewise, the centrifuge, calipers, and water bath for "tempering" the test are the same. The sample is prepared as directed on p. 242 of *Official Methods of Analysis*, 8th Ed., which specifies adjusting the temperature of the milk to 20°C. before sampling. The test is conducted as follows:

Part A.—With 17.6 ml pipet, transfer 18.0 g prepared sample to milk test bottle. Blow out milk in pipet tip after free outflow has ceased. Add 5 ml reagent (a) portionwise so as to wash all traces of milk into bulb. Shake to mix. Transfer bottle to boiling water bath, with level of water covering level of milk in bottle. (For comfort, a low bath is suggested, as a bath with high sides may direct steam from boiling water onto hands.) After bottle has been in bath ca 2 min., shake to remix raised cream and replace in bath for an additional 5 min. Shake to remix. Replace in bath for 5 more min. Shake to remix. Allow to remain in bath for a final 5 min.; then remove from bath without remixing contents. (Exceeding the specified heating time and exact timing of the shaking are not critical except that the bottles should not be heated longer than one-half hour, and they should remain in boiling water for at least 5 min. after the last shaking.)

Part B.—While bottle is still hot add 50% methyl alcohol to bring level of contents to top of graduated scale, allowing alcohol to flow down side of neck. If several tests are being conducted, remove one bottle at a time from boiling water, completing addition of alcohol solution before removing next bottle. Transfer bottle, while still

¹ Triton X-100 (Rohm & Haas Co.) and Tergitol Dispersant NPX (Carbide & Carbon Chemicals Co.) were found suitable and were used in these experiments. Many other detergents were investigated and were found unsuitable. There may be other suitable detergents that were not tried.

hot, to unheated centrifuge and whirl 2 min. Transfer bottle to water bath at 55–60°C., immerse it to level of top of fat column, and leave until column attains temperature of bath (ca 5 min.). Remove bottle from bath, wipe it, and with dividers or calipers measure fat column, in terms of % by weight, from lower surface to highest point of upper meniscus.

DPS DETERGENT METHOD FOR CREAM

PROCEDURE

Procedure with standard Babcock bottles.—Reagents (a) and (b) are the same as for milk. The test bottle and other apparatus, preparation of sample, and weighing of sample are the same as in the official Babcock cream test, *Official Methods of Analysis*, 8th Ed., 15.52 and 15.62–15.63.

Weigh a 9.0 g sample into cream test bottle. Add 10 ml reagent (a) portionwise so as to wash all traces of cream into bulb, shake to mix, and proceed as in the detergent method for milk, *Part A*, beginning "Transfer bottle to boiling water bath." In testing samples containing 40–50% fat, add 15 ml reagent (a). In testing samples whose approximate compositions are unknown, add 10 ml reagent (a) at beginning as usual, and then if sharp separation of a layer of some butter oil has not occurred after heating 7–10 min., add another 5 ml and mix. Continue in the usual manner, completing heating in boiling water for another 10 min. and remixing sample after the first 5 min. Measure fat column bottom to bottom.* Subtract 0.75% from the observed butterfat reading to obtain results, in % by weight, to conform more closely with Babcock test results.

Procedure with silicone-treated bottles.—Proceed as above, using Babcock bottles treated with silicone (General Electric Dri-Film, 9987, or equivalent) by saturating cloth with compound and rubbing interior of the neck of the clean, dry bottle with the saturated cloth until oily film is formed. Then rub with dry cloth until surface is clear. Avoid contaminating bulb of bottle with silicone, since longer centrifuging (5 min.) would be required to obtain all the fat. Avoid contact with compound. The silicone film is stable toward neutral and acid cleaners but is removed by strong alkalis.

No correction factor is required when silicone-treated bottles are used.

DPS DETERGENT METHOD FOR ICE CREAM

PROCEDURE

Reagents (a) and (b) are the same as for milk. Melt sample at room temperature or in a water bath at 40–50°C. and mix thoroughly with a rod or spoon. If sample contains insoluble particles, they may be dispersed before weighing by treatment with a high-speed stirrer with cutting blades, e.g., a Waring blender; alternatively, melted sample may be strained through a fine-mesh wire gauze and a correction calculated for weight of material removed. Weigh 9.0 g into ice cream test bottle and proceed as in the detergent method for milk, beginning under *Part A*, "Add 5 ml of reagent (a)." Measure fat column, in terms of % by weight, bottom to bottom (see footnote 2).

RESULTS AND DISCUSSION

Defects in the Original BDI Test.—No difficulty was experienced generally in applying the original detergent milk test (4) to samples of fresh whole milk (raw, pasteurized, or homogenized), plain ice cream mix, vanilla ice cream, and most samples of chocolate ice cream. Pearce (3),

* Glymol or red reader may be used to flatten the meniscus. Results with reader are usually slightly higher than readings bottom to bottom.

in a statistical study of his results, reported that the original BDI method was equivalent in accuracy to the official Babcock procedure when raw whole milk of normal fat content was tested. Values obtained in these laboratories with the original test on the products mentioned above checked closely, within experimental error, the values obtained with the Roesse-Gottlieb and the Babcock tests. However, an accurate test could not be obtained when 5 ml of reagent (a) was used, as in the original test, on samples of one particular brand of chocolate ice cream; the contents of the test bottle gelled upon heating and the resulting fat column contained considerable unreacted cream.

In addition to the failure of the milk test on one brand of chocolate ice cream, the following minor defects in the original test were occasionally noted: (1) Haziness in the solution just below the fat column, a very minor defect, not interfering with the accuracy of the test but sometimes obscuring the lower meniscus so as to make it necessary to use a strong light to read the test; (2) fuzziness at the bottom of the fat column, caused by the presence of cream-like emulsion particles, which destroyed the sharp outline of the meniscus and interfered slightly with the accuracy of the test; and (3) formation of coagulated gel, a more serious defect since sometimes this coagulum would float into the butterfat column, destroying the regularity of the lower meniscus and slightly changing the volume of the fat column.

Eliminating Defects from the Original BDI Test.—The presence of fuzziness was eliminated by making sure that the samples were heated for the full, specified period of time in boiling water and that they were centrifuged before they cooled too much. To insure adequate heating, slight changes were made in the heating and shaking procedure. The samples were shaken after 2 minutes in the bath rather than after 5 minutes. This effected more rapid distribution of heat within the bottle and also prevented coagulation of cream on the sides of the bottle at the surface before the proteins were completely dispersed. The samples were then left in boiling water for 15 minutes more and were shaken twice during that time. The original milk-test method specified only one shaking.

Investigation indicated that the reason for the failure of the original test on the one particular brand of chocolate ice cream was its unusually high solids-not-fat content and that apparently complete separation of the fat could be effected either by increasing the amount of detergent or of reagent (a) in the test or, preferably, by the addition of 2 per cent sodium bicarbonate to reagent (a). The addition of sodium bicarbonate also helped to eliminate the haziness below the fat column and the formation of coagulated gel.

Use of Sodium Bicarbonate.—Sodium bicarbonate apparently eliminates haziness below the fat column partly by a salting-out effect, which causes the sedimentation of cloudy material and increases the clearness of the

alcohol-water layer immediately below the fat column; thus a sharp, shiny lower meniscus is obtained and the appearance of the test is improved. In the modification in which the centrifuge is not used (4), however, this sedimentation effect tends to prevent some of the smaller fat globules from rising, and therefore the original reagent (a) without bicarbonate should be used for tests in which the sample is not centrifuged.

The effect of sodium bicarbonate in eliminating gel formation seems to be partly a neutralization effect, since the results suggested that gel formation was correlated with slight deterioration or slight souring of the samples. Sodium bicarbonate neutralizes any trace of acid phosphate which may be present as an impurity in solid sodium tetraphosphate; it also increases the stability of the aqueous solution.

The above advantages indicated that a test with sodium bicarbonate in reagent (a) could be applied, without an increase in amount of detergent or of reagent, to different types of samples of milk, ice cream, and other fluid dairy products containing up to about 15 per cent of butterfat,³ and would function effectively and accurately. As stated previously (4), fluid products containing more than about 15 per cent of butterfat require only the addition of more reagent.

Other Changes in the Original Test.—Pearce (3) found that reagent (a) was stable for at least a month at room temperature. Our experiments have confirmed this fact for both the regular reagent and the modified reagent containing bicarbonate; it has also been shown that both reagents are stable for at least a year in a refrigerator at about 10° C.

A "tempering" period of 5 rather than 15 minutes was found to be sufficient in the modification in which the centrifuge is used.

Miscellaneous Physico-Chemical Data.—Effects of pH were investigated in milk tests in which small amounts of normal acid or alkali were added to tests containing the sample and original reagent (a). With the pH below 6.5, the test was marred by precipitation in the contents of the bottle and incomplete separation of the fat; with the pH between 6.5 and 7.5, the fat separated sharply, clearly, and accurately; with the pH between 7.5 and 9.0, increasing fuzziness appeared in the fat column with increases in pH; with the pH above 9.0, considerable unreacted cream remained and appeared in the neck of the bottle upon centrifuging. In tests on fresh whole milk, pH values obtained with the original reagent were about 7.3 and with the reagent containing bicarbonate about 7.5. In tests on fresh cream, pH values of about 8.0 were obtained with 5, 10, or 15 ml of the reagent containing bicarbonate. Although pH 8.0 is slightly above the optimal pH of 6.5–7.5 mentioned above, this pH factor did not cause any difficulty in testing cream.

The detergent test, regular or modified, was not affected by the presence

³ Application of the detergent test to skim milk requires further modification of the method, not described in this paper.

of small amounts of mercuric chloride or potassium dichromate used as preservatives.

Accuracy of the Modified Detergent Test

(1) *Milk*.—Table 1 presents results on 44 samples of fresh whole milk. The average values obtained with the modified detergent test and with the Babcock test were identical. The maximum deviation between methods was 0.10 per cent and the average deviation was 0.03 per cent. The maximum deviation between duplicates in both methods was 0.06 per cent.

(2) *Cream*.—Table 2 presents results on 24 samples of fresh cream. The maximum deviation between duplicates in both methods was 0.5 per cent. The average obtained with the modified detergent test read bottom to bottom was 0.71 per cent higher, and bottom to reader was 0.80

TABLE 1.—Results obtained on milk with the DPS detergent test and the Babcock test

NO.	NUMBER ^a AND BREED OF COWS	BUTTERFAT		NO.	NUMBER ^a AND BREED OF COWS	BUTTERFAT	
		DPS TEST ^b	BABCOCK TEST ^b			DPS TEST ^b	BABCOCK TEST ^b
		<i>per cent</i>	<i>per cent</i>			<i>per cent</i>	<i>per cent</i>
1	15 Hol., Jer.	5.00	5.00	23	9 Ayrshire	3.85	3.87
2	7 Hol., Jer.	3.75	3.74	24	2 Guernsey	4.40	4.43
3	14 Hol., Jer.	4.15	4.13	25	8 Ayr., 1 Guer.	4.23	4.22
4	7 Hol., Jer.	3.38	3.42	26	1 Guernsey	6.39	6.40
5	14 Hol., Jer.	4.40	4.39	27	10 Ayrshire	3.87	3.87
6	7 Hol., Jer.	4.23	4.19	28	4 Guernsey	4.35	4.39
7	10 Hol., Jer.	3.39	3.41	29	6 Ayrshire	4.44	4.37
8	14 Hol., Jer.	4.34	4.32	30	2 Guernsey	6.21	6.14
9	6 Hol., Jer.	5.12	5.13	31	9 Ayrshire	4.44	4.43
10	10 Hol., Jer.	4.20	4.26	32	3 Guernsey	5.85	5.79
11	1 Holstein	3.31	3.28	33	10 Mixed	4.52	4.53
12	1 Jersey	5.92	5.91	34	35 Mixed	3.48	3.50
13	1 Jersey	5.91	5.97	35	300 Mixed	4.11	4.05
14	18 Mixed	4.49	4.45	36	10 Mixed	3.84	3.84
15	30 Mixed	3.97	3.93	37	1 Guernsey	5.40	5.30
16	38 Mixed	4.23	4.21	38	1 Guernsey	5.40	5.36
17	43 Mixed	3.70	3.65	39	1 Holstein	2.70	2.77
18	20 Mixed	3.21	3.27	40	1 Ayrshire	3.22	3.25
19	15 Mixed	3.30	3.37	41	1 Ayrshire	4.69	4.68
20	28 Mixed	5.23	5.18	42	1 Guernsey	7.95	7.93
21	33 Mixed	5.13	5.12	43	1 Holstein	2.90	2.97
22	360 Mixed	3.80	3.82	44	1 Holstein	3.57	3.62
Average:						4.41	4.41

^a Estimated from the volume of milk when not known precisely.

^b Tests conducted in Babcock test bottles especially selected for accuracy of calibration, tolerance limit 0.02%. All values averages of duplicates. Readings bottom to top.

TABLE 2.—Results obtained on cream with the DPS detergent, Babcock, and Roese-Gottlieb tests^a

NO.	DPS DETERGENT TEST ^b						BABCOCK TEST, ^b BOTTOM TO READER	ROESE-GOTTLIEB TEST
	BOTTOM TO BOTTOM			BOTTOM TO READER				
	OBSERVED	CORRECTED ^c	SILICONE TREATED	OBSERVED	CORRECTED ^c	SILICONE TREATED		
	per cent	per cent	per cent	per cent	per cent	per cent	per cent	per cent
1	19.0	18.0		19.3	18.3		18.5	18.43
2	30.1	29.1		30.2	29.2		29.1	28.60
3	27.3	26.3		27.3	26.3		26.3	25.83
4	37.0	36.0		37.0	36.0		35.7	35.17
5	20.5	19.5		20.6	19.6		19.5	19.61
6	29.1	28.1		29.1	28.1		28.0	27.95
7	20.0	19.0	19.4	20.1	19.1	19.5	19.4	19.16
8	28.7	27.7	28.2	28.9	27.9	28.1	28.3	27.57
9	41.1	40.1	40.3	41.2	40.2	40.2	40.3	39.99
10	23.7	22.7	23.5	23.8	22.8	23.4	23.5	22.95
11	36.3	35.3	36.0	36.3	35.3	36.0	35.9	35.40
12	49.8	48.8	48.9	49.7	48.7	48.9	49.1	48.66
13	20.2	19.2	19.4	20.2	19.2	19.3	19.3	19.30
14	30.3	29.3	29.5	30.4	29.4	29.6	29.6	29.39
15	40.9	39.9	40.3	40.8	39.8	40.3	40.2	40.00
16	21.1	20.1	20.4	21.4	20.4	20.6	20.8	20.64
17	31.5	30.5	31.1	31.7	30.7	31.1	30.9	30.78
18	43.1	42.1	42.5	43.2	42.2	42.5	42.5	41.94
19	19.8	18.8	19.3	20.1	19.1	19.4	19.4	19.49
20	30.6	29.6	29.9	30.7	29.7	29.9	30.3	29.89
21	41.5	40.5	41.0	41.5	40.5	41.0	40.5	40.52
22	23.0	22.0	22.5	23.1	22.1	22.5	22.5	22.43
23	35.3	34.3	34.5	35.5	34.5	34.5	34.7	34.45
24	47.4	46.4	46.5	47.5	46.5	46.3	46.0	46.00
Av. of 24	31.14	30.14		31.23	30.23		30.43	30.17
Av. of 18	32.46	31.46	31.84	32.56	31.56	31.84	31.84	31.59

^a All values averages of duplicates.

^b Tests conducted in Babcock cream test bottles especially selected for accuracy of calibration, tolerance limit 0.10%.

^c Actual reading minus factor 1.0%.

per cent higher than with the Babcock test; the average with the Babcock test read bottom to reader was 0.26 per cent higher than with the Roese-Gottlieb test. Accurate bottom-to-bottom readings with the Babcock test could not be made because of haziness in the fat columns.

The apparent reason for the indicated discrepancy between the results of the detergent test and of the Babcock test was found to be the greater curvature of the bottom meniscus in the detergent test; the bottom meniscus in the Babcock test was flat, as shown in Fig. 1. This finding was

indicated by results of detergent tests in which the last 18 samples in Table 2 were also tested in bottles the necks of which had been coated with the silicone film, as described previously (4), to flatten the curved bottom meniscus. The average values obtained with the detergent test conducted in silicone-treated bottles and with the Babcock test were identical, 31.84 per cent, when both tests were read bottom to reader. The Roese-Gottlieb test average on these 18 samples was 31.59 per cent.

The apparent reason for the indicated discrepancy between the Babcock and Roese-Gottlieb test results, averaging 0.26 per cent as indicated in Table 2, may be the presence of impurities in the Babcock test fat. Similarly, the presence of impurities in the detergent test fat is indicated by the close agreement of results by the Babcock test and by the detergent test in silicone-treated bottles.

The above results indicated that detergent test readings on cream, either bottom to bottom or bottom to reader, if obtained with silicone-treated bottles, checked closely without correction with Babcock test readings and would conform more closely with Roese-Gottlieb test results if corrected, for practical purposes, by a factor of minus 0.25 per cent; and, if obtained with regular test bottles, they would conform more closely with Babcock test readings if corrected by a factor of minus 0.75 per cent and with Roese-Gottlieb test results if corrected by minus 1.0 per cent.

Corrected by minus 1.0 per cent, as shown in Table 2, the average of the detergent test results read bottom to bottom was 30.14 per cent, and read bottom to reader, 30.23 per cent; these results check closely with 30.17 per cent, the Roese-Gottlieb test average. Deviations of the individual detergent test values, corrected by minus 1.0 per cent, from the Roese-Gottlieb test values in Table 2 were: Read bottom to bottom, maximum 0.83 per cent and average 0.28 per cent; read bottom to reader, maximum 0.83 per cent and average 0.23 per cent.

Concerning the nature of the impurities in the detergent test fat, results of a few preliminary analyses on fat separated in milk and cream tests, with Triton X-100 and also with Tergitol Dispersant NPX, indicated that the fat contained, in per cent by weight, approximately 1.7 per cent of detergent and 1.0 per cent of volatile substances, such as water, alcohol, and other volatile impurities. Slight amounts of other non-volatile impurities may also be present.

(3) *Ice Cream*.—Table 3 presents results of experiments on five different brands of vanilla ice cream (No. 1-5) and two different brands of chocolate ice cream (No. 6 and 7). Sample No. 7 is the brand which could not be tested by the original method for milk. The results indicate that the values obtained with the modified test, using either Triton X-100 or Tergitol Dispersant NPX, agreed closely, within experimental error, with those obtained by the Roese-Gottlieb test.

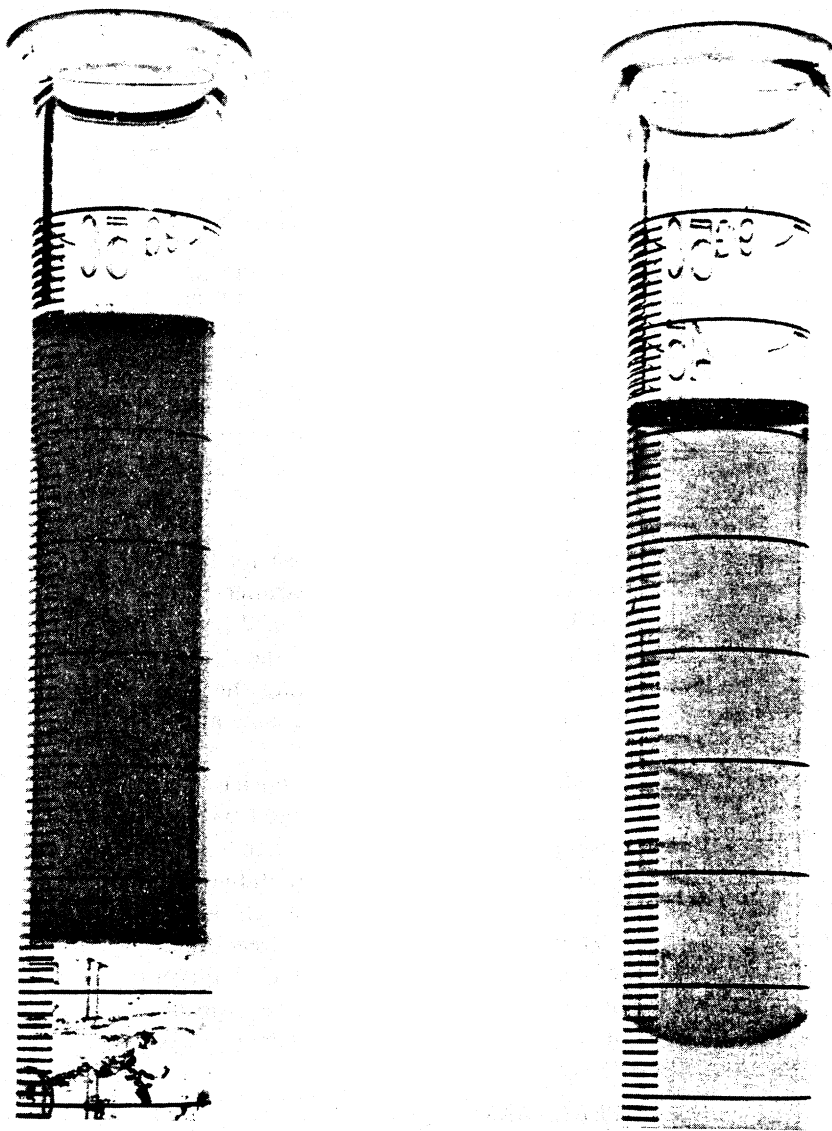


FIG. 1.—Fat columns in Babcock test (left) and detergent test (right) on a sample of cream. Note flat bottom meniscus, indistinct upper meniscus, hazy fat column, and charred particles in Babcock test; curved bottom meniscus, distinct upper meniscus, and clear fat column in detergent test.

TABLE 3.—Results obtained on ice cream with the DPS detergent test and the Roese-Gottlieb test^a

NO.	DETERGENT TEST ^b WITH TRITON X-100		DETERGENT TEST ^b WITH TERGITOL DISPERSANT NPX		ROESE-GOTTLIEB TEST
	BOTTOM TO BOTTOM	BOTTOM TO READER	BOTTOM TO BOTTOM	BOTTOM TO READER	
	<i>per cent fat</i>	<i>per cent fat</i>	<i>per cent fat</i>	<i>per cent fat</i>	<i>per cent fat</i>
1	12.2	12.2	12.1	12.1	12.35
2	12.2	12.2	12.2	12.2	12.39
3	11.5	11.5	11.3	11.3	11.23
4	12.1	12.2	12.2	12.2	11.87
5	12.5	12.5	12.3	12.4	12.38
6	12.0	12.0	12.1	12.2	12.00
7	12.2	12.2	12.0	12.1	12.10
Av.	12.10	12.11	12.03	12.07	12.05

^a All values averages of duplicates.

^b Tests conducted in Babcock ice cream test bottles especially selected for accuracy of calibration, tolerance limit 0.04%.

SUMMARY

Changes have been made in the original BDI detergent test for butterfat (4), improving the methodology, accuracy, and appearance of the test and increasing its usefulness. Changes include addition of 2 per cent of sodium bicarbonate to the detergent-tetraphosphate reagent, slight changes in the heating and shaking procedure, and shortening the "tempering" period. The modified procedure for whole milk, cream, and ice cream is presented.

Fat values obtained with the modified test on milk and on ice cream agreed closely, within experimental error, with those obtained with the Babcock and the Roese-Gottlieb tests, respectively. Detergent test results on cream averaged approximately 0.75 per cent higher than Babcock results and 1.0 per cent higher than Roese-Gottlieb results. The curved bottom meniscus in the detergent test on cream was found to account for the increase in reading over that of the Babcock test. Results of detergent tests on cream, conducted in silicone-treated bottles to flatten the bottom meniscus, averaged the same as Babcock test results and averaged 0.25 per cent higher than Roese-Gottlieb results.

REFERENCES

- (1) *Official Methods of Analysis*, 8th Ed., Association of Official Agricultural Chemists, Box 540, Benjamin Franklin Station, Washington 4, D. C., 1955.
- (2) *Instruction Manual for Setting Up and Operating the Mojonnier Milk Tester*, Mojonnier Bros. Co., Chicago, Ill., 1925.
- (3) PEARCE, S. J., *Factors Affecting the Breaking of Milk and Cream Emulsions by Surface-active Agents*. Thesis (M.S.). Oregon State College, Corvallis, 1953.
- (4) SAGER, O. S., and SANDERS, G. P., *Milk Ind. Found. Conv. Proc.*, Lab. Sec., 4, 29 (1952).